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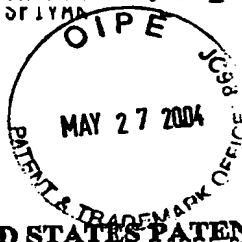
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URICOH-RTR SYH3MD  
OBLON SYH3MD

NO. INO. 0077r. P. 2



DOCKET NO: 197484US23

**IN THE UNITED STATES PATENT & TRADEMARK OFFICE**

IN RE APPLICATION OF

YASUO SUZUKI, ET AL.

SERIAL NO: 09/679,480

FILED: OCTOBER 5, 2000

FOR: ELECTROPHOTOGRAPHIC  
PHOTORECEPTOR AND  
ELECTROPHOTOGRAPHIC IMAGE  
FORMING METHOD AND APPARATUS  
USING THE PHOTORECEPTOR

:

: EXAMINER: DOTE, J.

:

: GROUP ART UNIT: 1756

:

**DECLARATION UNDER 37 C.F.R. § 1.132**

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Yasuo Suzuki who deposes and states:

1. That I am a graduate of Tohoku University and received a masters degree in the year 1987.

2. That I have been employed by Ricoh Co., Ltd., for 17 years as a manager in the field of electrophotography.

3. That I understand the English language or, at least, that the contents of the Declaration were made clear to me prior to executing the same.

4. That the following experiment was carried out by me or under my direct supervision and control.

#### 4.1 Objective

Repeating Example 5 and Comparative Example 5 of the present application to show that the present invention does not depend on the thickness of the intermediate layer and the drum diameter.

#### 4.2 Experiment

Example 5 and Comparative Example 5 of the present application were repeated while the intermediate layer thickness and the drum diameter were changed to 4.5  $\mu\text{m}$  and 80 mm, respectively, and a digital copier IMAGIO MF530 manufactured by Ricoh Co., Ltd, was used as an evaluation machine. The experiment was as follows.

The following photoconductors of Examples A, B and C in each of which an organic sulfur-containing antioxidant was included, and a photoconductor of Comparative Example A in which no organic sulfur-containing antioxidant was included, were prepared.

##### Substrate used

An aluminum substrate having a diameter of 80 mm and a length of 359 mm.

##### Preparation of intermediate layer

The following components were mixed and dispersed for 72 hours using a ball mill, to prepare an intermediate layer coating liquid.

Titanium dioxide	70 parts by weight (CR-EL from Ishihara Sangyo Kaisha Ltd.)
Alkyd resin	15 parts by weight (BEKKOLITE M6401-50-S from Dainippon Ink and Chemicals, Inc., solid content of 50 %)
Melamine resin	10 parts by weight (SUPER BEKKAMIN L-121-60 from Dainippon Ink and Chemicals, solid content of 60 % by weight)
Methyl ethyl ketone	100 parts by weight

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The thus prepared intermediate layer coating liquid was coated on the surface of the aluminum drum mentioned above, and dried for 20 minutes at 130°C to form an intermediate layer having a dry thickness of 4.5  $\mu\text{m}$ .

#### Preparation of charge generation layer

Four parts by weight of an asymmetric disazo pigment having a formula (VI) and 3.0 parts by weight of a  $\pi$ -form metal-free phthalocyanine were added to a resin solution which had been prepared by dissolving 1.4 parts by weight of polyvinyl butyral (S-LEC BM-S from Sekisui Chemical Co., Ltd.) in 150 parts by weight of cyclohexanone. The mixture was dispersed for 72 hours using a ball mill. After dispersion, 210 parts by weight of cyclohexanone were added thereto and the mixture was dispersed for 3 hours, to prepare a charge generation layer coating liquid.

The coating liquid was coated on the intermediate layer, followed by drying for 10 minutes at 130°C. Thus, a charge generation layer having a thickness of 0.20  $\mu\text{m}$  was prepared.

#### Preparation of charge transport layer

At first, 7.5 parts by weight of a charge transport material having a formula (VI), 10 parts by weight of a polycarbonate resin (Z-form, viscosity average molecular weight of 40,000), and 0.002 parts of a silicone oil (KF-50 from Shin-Etsu Chemical Co., Ltd.), optionally together with 0.07 parts by weight of sulfur-containing antioxidant, were dissolved in 100 parts by weight of toluene, to prepare a charge transport layer coating liquid.

The thus prepared charge transport layer coating liquid was coated on the charge generation layer, followed by drying for 25 minutes at 130°C. Thus, a charge transport layer having a thickness of 28  $\mu\text{m}$  was prepared.

Thus, photoconductors of Examples A, B and C and Comparative Example A were prepared.

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CRICOH-RTR SYH

NO. 0077 P. 55

Example	Sulfur-containing antioxidant
A	S-1
B	(III-3)
C	(III-6)
Comparative Example A	No

Each of the thus prepared photoconductors was set in the digital copier to be evaluated. The evaluation items and methods are as follows.

(1) Light resistance ( $\Delta$  VD)

The light resistance was evaluated in the same way as described in the present application. Namely, each photoconductor was set in the copier to measure the potential (VD, units of -V) of a non-lighted portion of the photoconductor. Then the photoconductor was allowed to settle for 60 minutes under a white fluorescent lamp such that the illuminance on the photoconductor is 1000 lux. Then the photoconductor which was exposed to light was set again in the copier to measure the potential (VD', units of -V) of a non-lighted portion thereof. The light resistance (i.e.,  $\Delta$  VD) of the photoconductor is defined as (VD - VD').

In this regard, the potentials (VD and VD') were measured by a potential meter set at the developing portion while a filter with ND of 0.5 was attached to the light irradiator to reduce the light quantity to one half (1/2).

In addition, the initial potential (i.e., VD) was controlled so as to be about -900 V by controlling the applied voltage of the charging member.

(2) Image qualities

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NO. 0077 P. 6

A running test in which 200,000 copies are continuously produced using a recording paper was performed on each photoconductor under conditions of 25°C and 50 % RH using an original including a black solid portion and having an image area proportion of 5 %. In the running test, the copies were visually observed to determine the number of copies in which black spots having a diameter not less than 0.1 mm are present in white areas with a frequency of 1 piece/cm<sup>2</sup> or more, and to determine whether the image density decreases and undesired images are present therein.

#### 4.3 Results

The results are shown in Table A.

Table A

	Drum diameter (mm)	Intermediate layer thickness ( $\mu$ m)	$\Delta$ VD (V)	Black spots observed from Copy #	Undesired Images
Ex. 4 (from page 43 of application)	30	3	80	45,000	no
Ex. 5 (from page 46 of application)	30	3	50	0	no
Ex. A	80	4.5	50	173,000	No
Ex. B	80	4.5	20	0	No
Ex. C	80	4.5	20	0	No
Comp. Ex. A	80	4.5	95	105,000	Background fouling

As can be understood from Table A, it is clear that the photoconductor of the present invention is superior to the comparative photoconductor. Further, it has been demonstrated that this superior result is achieved independent of the drum diameter and the intermediate layer thickness. For comparison the data for Examples 4 and 5 of the specification using a drum diameter of 30 mm and a thickness of the intermediate layer of 3  $\mu$ m are shown in the Table A. In addition, Examples A, B and C are superior to Comparative Example A which uses the photoreceptor of JP'998 (JP '998 compound (I)-24 which is Formula (VII) of the present invention). Notably, background fouling occurs in Comparative Example A and black spots appear after 105,000 copies. The unexpected superior results are not disclosed or suggested by JP '998, JP '250, Kanoto, Kakuta, or DERWENT Abstract '039, alone or in combination.

### **5.1 Objectives**

(1) The compound (I)-29 used in Example 9 of JP07-128890 (JP '890) is the same as the compound VIII used in Comparative Examples 13 to 16 in the present application. The specification shows data of compound VIII in combination with  $\tau$ -form metal-free phthalocyanine. This Declaration provides data for a combination of the compound VIII with X-form metal-free phthalocyanine.

(2) This Declaration provides data for a combination of compound (I)-30 used in Example 10 of JP '890 with X-form metal-free phthalocyanine.

### **5.2 Experiment**

Applicants have prepared various photoreceptors and evaluated the photoreceptors in the same way as described in the present application except that the materials were changed.

#### **Preparation of intermediate layer**

The following components were mixed and dispersed for 72 hours using a ball mill to prepare an intermediate layer coating liquid.

Titanium oxide 70 parts by weight

(CR-EL from Ishihara Sangyo Kaisha Ltd.)

Alkyd resin 15 parts by weight

(BEKKOLITE M-6401-50-S from Dainippon Ink & Chemicals Inc., solid content of 50%)

Melamine resin 10 parts by weight

(SUPER BEKKAMIN L-121-60 from Dainippon Ink & Chemicals Inc., solid content of 60%)

Methyl ethyl ketone 100 parts by weight

The thus prepared intermediate layer coating liquid was coated on the peripheral surface of an aluminum drum having a diameter of 30 mm and a length of 340 mm, followed by drying at 130°C for 20 minutes, to prepare an intermediate layer having a thickness of 3.0  $\mu\text{m}$ .

#### Preparation of charge generation layer

Four (4.0) parts by weight of an asymmetric disazo pigment (see Table B) and 3.0 parts by weight of X-form metal-free phthalocyanine pigment were added to a resin solution which had been prepared by dissolving 1.4 parts by weight of a polyvinyl butyral (S-LEC BM-S from Sekisui Chemical Co., Ltd.) in 150 parts by weight of cyclohexanone. The mixture was subjected to a dispersing treatment for 72 hours using a ball mill. Then the dispersion was mixed with 210 parts by weight of cyclohexanone and the mixture was further dispersed for 3 hours to prepare a charge generation layer coating liquid.



The thus prepared charge generation layer coating liquid was coated on the intermediate layer prepared above, followed by drying at 130°C for 10 minutes, to prepare a *charge generation layer having a thickness of 0.20  $\mu\text{m}$ .*

Preparation of charge transport layer coating liquid

Seven point five (7.5) parts by weight of a charge transfer material having formula (VI), 10 parts by weight of a polycarbonate (Z-form polycarbonate having a viscosity average molecular weight of 40,000), 0.07 parts by weight of an additive (see Table B), and 0.002 parts by weight of a silicone oil (KF-50 from Shin-Etsu Chemical Co., Ltd.) were dissolved in 100 parts by weight of toluene to prepare a charge transfer coating liquid. The thus prepared charge transport layer coating liquid was coated on the charge generation layer prepared above, followed by drying at 135°C for 25 minutes, to prepare a charge transport layer having a thickness of 28  $\mu\text{m}$ .

Thus, the photoreceptors, details of which are shown in Table B, were prepared.

The photoreceptors were evaluated in the same way as described in the specification of the present application.

5.3 Results

The results are shown in Table B.

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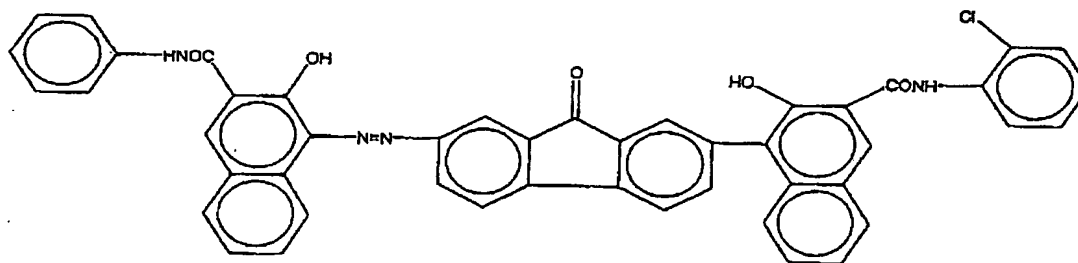
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NO. 0077 P. 10

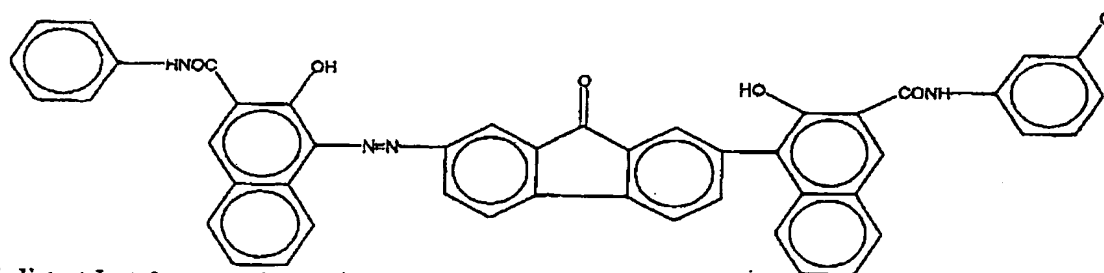
Table B

	Asymmetric disazo pigment	Additive	$\Delta$ VD	Black spots observed from Copy #	Undesired images
Example D	Formula (VIII)*	(III)-3	20	0	No
Example B	Formula (VIII)*	(III)-5	20	0	No
Example F	Formula (VIII)*	(III)-6	20	0	No
Example G	Formula A**	(III)-3	15	0	No
Example H	Formula A**	(III)-5	20	0	No
Example J	Formula A**	(III)-6	20	0	No
Comp. Ex. B	Formula (VIII)*	No	105	36,000	Background fouling
Comp. Ex. C	Formula (VIII)*	(1)* <sup>3</sup>	90	40,000	No
Comp. Ex. D	Formula (VIII)*	(2)* <sup>4</sup>	95	39,000	No
Comp. Ex. E	Formula (VIII)*	(3)* <sup>5</sup>	105	36,000	No
Comp. Ex. F	Formula A**	No	100	36,000	Background fouling
Comp. Ex. G	Formula A**	(1)* <sup>3</sup>	90	39,000	No
Comp. Ex. H	Formula A**	(2)* <sup>4</sup>	90	38,000	No
Comp. Ex. J	Formula A**	(3)* <sup>5</sup>	100	36,000	No

\*Formula (VIII) (same as (I)-29 in JP '890)



\*\*Formula (A) (same as (I)-30 in JP '890)



\*3: 2,6-di-tert-butyl-p-cresol

\*4: 4,4'-butylidenebis(6-tert-butyl-3-methylphenol)

\*5: tris(2,4-di-tert-butylphenyl)phosphite

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NO. 0077, P. 11

The Examples according to the present invention that use 1) a combination of the compound VIII with X-form metal-free phthalocyanine and a sulfur-containing antioxidant or 2) a combination of compound (I)-30 used in Example 10 of JP '890 with X-form metal-free phthalocyanine and a sulfur-containing antioxidant do not exhibit black spots or undesired images (Examples D-F and G-J). In contrast, if no sulfur-containing antioxidant is used, black spots and undesired images occur (Comparative Examples B and F). If other additives are used, the superior properties of the present invention cannot be achieved (Comparative Examples C-E and G-J). Thus, the superior results of the present invention have been demonstrated.

These superior results are not disclosed or suggested by JP '890, JP '250, Kanoto, or U.S. 3,357,989 (Byrne), alone or in combination.

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

7. Further deponent saith not.

Yasuo Suzuki  
Signature

May 21, 2004  
Date